

S507883

(continued)  
the ring substituents to a polymer chain. The new method is being used to determine the molecular weight of the polymer.

## Test Report

### Measurement of the molecular weights

CBI

CBI

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**[Summary]**

The measurements of the average molecular weights and the contents of components having molecular weight of less than 1000 and less than 500 of the test sample [CBI] were carried out using GPC. The results are shown in Table A.

Table A Average molecular weights and  
Contents of components less than MW 1000 and less than MW 500\*

Number average molecular weight ( $M_n$ )	[CBI]
Weight average molecular weight ( $M_w$ )	
Z average molecular weight ( $M_z$ )	
Polydispersity ( $M_w / M_n$ )	
Polydispersity ( $M_z / M_w$ )	
Content of component < MW 1000	
Content of component < MW 500	

\*relative values of polystyrene standards

**1. Sponsor**

Name  
Address

CBI

**2. Test Laboratory**

Name  
Address

CBI

**3. Objective**

The objective of this measurement is to obtain the average molecular weights and contents of components having molecular weight of less than 1000 and less than 500 of CBI

**4. Sample**

CBI

**5. Experimental****5.1 Principle**

Gel permeation chromatography (GPC) is a family of the liquid chromatography that separates molecules of the polymer sample by the difference in their molecular size. The polymer molecules are separated in the column packed with the gels that have the pore of the same size as the polymer size in dilute solution (hydrodynamic volume). The high molecular weight component is eluted faster than the low molecular weight component because the high molecular weight component is slightly permeable into the pore of the gel. Therefore GPC provides the molecular weight distribution curve and the average molecular weights of the polymer.

**5.2 Measurement Parameters**

Apparatus	Gel Permeation Chromatograph (GPC-19)
Detector	Differential refractive index detector RI-8020 (TOSOH)
Data Processing system	GPC Data Processing System (Toray research Center)
Column set	Column set of three TSKgel MultiporeH <sub>XL</sub> -M (TOSOH)
Mobile Phase	Tetrahydrofuran

CBI

Flow rate	1.0 mL/min
Temperature	23±2°C
Sample Concentration	0.20% (w/v)
Solubility	Soluble
Filtration	0.45 µm-Millex-LH (Millipore)
Injection volume	0.200 mL
the calibration curve of the standards	Polystyrene (PS) standards (TOSOH)

## 6. Results

### 6.1 GPC Curve

The GPC curve of the sample is shown in Fig.1 and Fig.2. The signal of the eluted sample was detected from about 18 min. to 33 min. The peaks detected after about 33 min. of the elution time were the solvent-composition-change, solvent-impurity and/or the system peak (It was confirmed by the GPC measurement of the solvent. See Fig.3).

### 6.2 Calibration curve of molecular weight

The calibration curve of polystyrene (PS) standards is shown in Fig.4. The calibration curve was obtained from the relationship between the molecular weight and elution time under the same GPC conditions. In this report the calculated molecular weights were relative values of PS standards.

$$\log M = A_0 + A_1 \cdot t \quad (1)$$

where,  $M$  : Molecular weight

$t$  : Elution time

$A_i$  : Coefficients

### 6.3 Calculation of molecular weight distribution (MWD) and average molecular weights

The MWD was originated by the RI curves of the sample (Fig.1 and Fig.2) and the calibration curve (Fig.4) using GPC data processing system. The MWD curve and the average molecular weights are shown in Fig.5, Fig.6 and Table A, respectively.

The average molecular weights were calculated according to Eqs. (2), (3) and (4).

$$M_n = \sum (N_i \cdot M_i) / \sum N_i \quad (2)$$

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$$M_W = \sum (N_i \cdot M_i^2) / \sum (N_i \cdot M_i) \quad (3)$$

$$M_Z = \sum (N_i \cdot M_i^3) / \sum (N_i \cdot M_i^2) \quad (4)$$

- where,
- $M_n$  : Number average molecular weight
  - $M_w$  : Weight average molecular weight
  - $M_z$  : Z average molecular weight
  - $N_i$  : molar fraction at elution time  $t_i$
  - $M_i$  : molecular weight at elution time  $t_i$

The polydispersity ( $M_w / M_n$ ,  $M_z / M_w$ ), indicating the index of the dispersion of MWD, is shown in Table A.

- where,  $M_w / M_n$  : Polydispersity  
 (the index of the dispersion in the low molecular weight region)  
 $M_z / M_w$  : Polydispersity  
 (the index of the dispersion in the high molecular weight region)

#### 6.4 Content of the components less than molecular weight 1000 and 500

The raw data of MWD calculated according to the section 6.3 are shown in Table 1 and Table 2. The content of the components having molecular weight of less than 1000 and less than 500 were CBI

#### 7. Conclusion

○ The measurements of the average molecular weights and the contents of components molecular weight of less than 1000 and less than 500 of CBI were carried out using GPC. The number average molecular weight and the weight average molecular weight of the sample were CBI respectively. The content of the components having molecular weight of less than 1000 and less than 500 were CBI

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This work was carried out by Chemist Chihiro Hamashige, Research Associate Kazutomo Akasaka and Manager Tsuneyuki Yamane at [REDACTED]

[REDACTED]  
CBI

Manager : Tsuneyuki Yamane

Signature : Tsuneyuki YamaneDate : Sep. 11, 2013

Study Director : Kazutomo Akasaka

Signature : Kazutomo AkasakaDate : Sep. 11, 2013

○

○

Table 1 Molecular Weight Data

Sample : L B C

File : A05.MWD

Area : 278.00 mV · sec

Operator: Hamashige

Date : 2013/9/4

Mn : [REDACTED]

Mw : [REDACTED]

Mz : [REDACTED]

Mv : [REDACTED]

Mw/Mn: [REDACTED]

Mz/Mw: [REDACTED]

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Retention Time (min)	Molecular Weight	Intensity (mV)	d(Weight) / d Log (MW)	Weight %
18.23	441128	0.000	0.000	100.00
18.32	422401	0.000	0.000	100.00
18.40	404469	0.002	0.002	100.00
18.48	387298	0.003	0.003	99.99
18.57	370856	0.003	0.003	99.99
18.65	355113	0.006	0.006	99.98
18.73	340038	0.006	0.006	99.97
18.82	325602	0.007	0.007	99.95
18.90	311780	0.009	0.008	99.94
18.98	298544	0.011	0.011	99.92
19.07	285870	0.014	0.013	99.90
19.15	273734	0.015	0.014	99.87
19.23	262114	0.018	0.017	99.84
19.32	250986	0.022	0.021	99.80
19.40	240331	0.025	0.024	99.76
19.48	230129	0.027	0.026	99.71
19.57	220359	0.031	0.029	99.66
19.65	211005	0.035	0.033	99.60
19.73	202047	0.040	0.039	99.53
19.82	193470	0.046	0.044	99.45
19.90	185256	0.050	0.048	99.37
19.98	177392	0.056	0.054	99.27
20.07	169861	0.063	0.060	99.16
20.15	162650	0.069	0.066	99.04
20.23	155745	0.077	0.073	98.91
20.32	149134	0.085	0.081	98.76
20.40	142803	0.092	0.088	98.60
20.48	136740	0.101	0.096	98.43
20.57	130935	0.111	0.106	98.24
20.65	125377	0.120	0.115	98.03
20.73	120054	0.131	0.125	97.80
20.82	114958	0.142	0.136	97.55
20.90	110078	0.154	0.147	97.28
20.98	105405	0.168	0.160	96.99
21.07	100930	0.181	0.173	96.67
21.15	96645	0.195	0.187	96.33
21.23	92542	0.211	0.201	95.96
21.32	88614	0.227	0.217	95.57
21.40	84852	0.243	0.232	95.14
21.48	81250	0.260	0.248	94.69
21.57	77800	0.278	0.266	94.20
21.65	74498	0.297	0.283	93.68

File : A05.MWD

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Retention Time (min)	Molecular Weight	Intensity (mV)	d(Weight) / d Log (MW)	Weight %
21.73	71335	0.316	0.302	93.13
21.82	68307	0.336	0.321	92.54
21.90	65407	0.358	0.341	91.91
21.98	62630	0.380	0.363	91.24
22.07	59971	0.402	0.384	90.53
22.15	57426	0.426	0.406	89.79
22.23	54988	0.449	0.429	88.99
22.32	52653	0.473	0.452	88.16
22.40	50418	0.498	0.475	87.28
22.48	48278	0.523	0.499	86.36
22.57	46228	0.548	0.524	85.39
22.65	44266	0.575	0.549	84.38
22.73	42387	0.602	0.575	83.32
22.82	40587	0.628	0.600	82.20
22.90	38864	0.656	0.626	81.04
22.98	37214	0.683	0.652	79.83
23.07	35634	0.710	0.677	78.58
23.15	34122	0.737	0.704	77.27
23.23	32673	0.765	0.731	75.92
23.32	31286	0.792	0.756	74.51
23.40	29958	0.819	0.782	73.06
23.48	28686	0.845	0.807	71.56
23.57	27468	0.871	0.831	70.01
23.65	26302	0.896	0.855	68.41
23.73	25186	0.919	0.878	66.78
23.82	24116	0.943	0.900	65.10
23.90	23093	0.964	0.920	63.38
23.98	22112	0.984	0.940	61.62
24.07	21174	1.003	0.957	59.83
24.15	20275	1.018	0.972	58.01
24.23	19414	1.032	0.985	56.17
24.32	18590	1.043	0.996	54.30
24.40	17801	1.051	1.004	52.41
24.48	17045	1.056	1.008	50.52
24.57	16321	1.058	1.010	48.62
24.65	15629	1.057	1.009	46.71
24.73	14965	1.054	1.006	44.82
24.82	14330	1.046	0.999	42.93
24.90	13721	1.035	0.988	41.06
24.98	13139	1.021	0.974	39.21
25.07	12581	1.005	0.959	37.39
25.15	12047	0.985	0.941	35.61
25.23	11536	0.965	0.921	33.86
25.32	11046	0.941	0.898	32.15
25.40	10577	0.915	0.874	30.48
25.48	10128	0.888	0.848	28.86
25.57	9698	0.859	0.820	27.30
25.65	9286	0.830	0.792	25.78
25.73	8892	0.799	0.763	24.33

File : A05.MWD

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Retention Time (min)	Molecular Weight	Intensity (mV)	$\frac{d(\text{Weight})}{d \log (\text{MW})}$	Weight %
25.82	8515	0.768	0.733	22.92
25.90	8153	0.738	0.704	21.57
25.98	7807	0.707	0.675	20.28
26.07	7476	0.675	0.645	19.04
26.15	7158	0.646	0.616	17.86
26.23	6854	0.615	0.587	16.73
26.32	6563	0.585	0.559	15.66
26.40	6285	0.556	0.531	14.64
26.48	6018	0.527	0.503	13.67
26.57	5762	0.499	0.477	12.75
26.65	5518	0.474	0.452	11.88
26.73	5284	0.448	0.428	11.05
26.82	5059	0.423	0.404	10.28
26.90	4845	0.399	0.381	9.54
26.98	4639	0.376	0.359	8.85
27.07	4442	0.355	0.338	8.19
27.15	4253	0.334	0.319	7.58
27.23	4073	0.312	0.298	7.00
27.32	3900	0.294	0.280	6.46
27.40	3734	0.274	0.262	5.95
27.48	3576	0.255	0.244	5.48
27.57	3424	0.239	0.228	5.04
27.65	3279	0.222	0.212	4.63
27.73	3139	0.206	0.197	4.25
27.82	3006	0.192	0.183	3.89
27.90	2879	0.177	0.169	3.56
27.98	2756	0.162	0.155	3.26
28.07	2639	0.151	0.144	2.98
28.15	2527	0.139	0.133	2.72
28.23	2420	0.129	0.123	2.49
28.32	2317	0.117	0.112	2.27
28.40	2219	0.107	0.102	2.07
28.48	2125	0.100	0.095	1.88
28.57	2035	0.091	0.087	1.71
28.65	1948	0.083	0.080	1.56
28.73	1865	0.077	0.073	1.42
28.82	1786	0.069	0.066	1.29
28.90	1710	0.063	0.060	1.17
28.98	1638	0.058	0.055	1.06
29.07	1568	0.052	0.050	0.96
29.15	1502	0.047	0.045	0.87
29.23	1438	0.043	0.041	0.79
29.32	1377	0.039	0.037	0.72
29.40	1318	0.035	0.034	0.65
29.48	1262	0.033	0.031	0.59
29.57	1209	0.029	0.028	0.54
29.65	1158	0.027	0.025	0.49
29.73	1108	0.024	0.023	0.44
29.82	1061	0.022	0.021	0.40

File : A05.MWD

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Retention Time (min)	Molecular Weight	Intensity (mV)	$\frac{d(\text{Weight})}{d \log (\text{MW})}$	Weight %
29.90	1016	0.020	0.019	0.37
29.98	973	0.018	0.017	0.33
30.07	932	0.016	0.016	0.30
30.15	892	0.016	0.015	0.27
30.23	854	0.014	0.013	0.25
30.32	818	0.013	0.013	0.22
30.40	783	0.014	0.013	0.20
30.48	750	0.013	0.012	0.18
30.57	718	0.013	0.013	0.15
30.65	688	0.014	0.013	0.13
30.73	659	0.013	0.013	0.10
30.82	631	0.012	0.011	0.08
30.90	604	0.010	0.009	0.06
30.98	578	0.007	0.007	0.05
31.07	554	0.006	0.005	0.04
31.15	530	0.004	0.004	0.03
31.23	508	0.001	0.001	0.02
31.32	486	0.002	0.002	0.02
31.40	465	0.002	0.002	0.02
31.48	446	0.001	0.001	0.01
31.57	427	0.001	0.001	0.01
31.65	409	0.000	0.000	0.01
31.73	391	0.000	0.000	0.01
31.82	375	0.000	0.000	0.01
31.90	359	0.000	0.000	0.01
31.98	344	0.000	0.000	0.01
32.07	329	0.001	0.001	0.01
32.15	315	0.000	0.000	0.01
32.23	302	0.001	0.001	0.01
32.32	289	0.002	0.002	0.00
32.40	277	0.001	0.001	0.00
32.48	265	0.001	0.001	0.00
32.57	254	0.000	0.000	0.00
32.65	243	0.000	0.000	0.00
32.73	233	0.000	0.000	0.00
32.82	223	0.000	0.000	0.00
32.90	213	0.000	0.000	0.00
32.98	204	0.000	0.000	0.00

File : A05.MWD

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Table 2 Molecular Weight Data

S507883

Sample : **L B C**  
 File : A06.MWD  
 Area : 289.85 mV · sec  
 Operator: Hamashige  
 Date : 2013/9/4  
 Mn : **CBI**  
 Mw :  
 Mz :  
 Mv :  
 Mw/Mn:  
 Mz/Mw:

Retention Time (min)	Molecular Weight	Intensity (mV)	$\frac{d(\text{Weight})}{d \log (\text{MW})}$	Weight %
18.23	441128	0.000	0.000	100.00
18.32	422401	0.001	0.001	100.00
18.40	404469	0.002	0.002	100.00
18.48	387298	0.003	0.003	99.99
18.57	370856	0.005	0.004	99.98
18.65	355113	0.005	0.005	99.98
18.73	340038	0.007	0.007	99.96
18.82	325602	0.009	0.008	99.95
18.90	311780	0.010	0.009	99.93
18.98	298544	0.014	0.013	99.91
19.07	285870	0.016	0.015	99.89
19.15	273734	0.019	0.017	99.86
19.23	262114	0.021	0.019	99.82
19.32	250986	0.024	0.022	99.78
19.40	240331	0.028	0.025	99.74
19.48	230129	0.032	0.029	99.69
19.57	220359	0.035	0.032	99.63
19.65	211005	0.039	0.036	99.56
19.73	202047	0.043	0.040	99.49
19.82	193470	0.049	0.045	99.41
19.90	185256	0.053	0.049	99.32
19.98	177392	0.060	0.055	99.23
20.07	169861	0.067	0.061	99.11
20.15	162650	0.074	0.068	98.99
20.23	155745	0.082	0.075	98.86
20.32	149134	0.090	0.083	98.71
20.40	142803	0.098	0.089	98.54
20.48	136740	0.107	0.098	98.36
20.57	130935	0.117	0.108	98.17
20.65	125377	0.127	0.116	97.96
20.73	120054	0.137	0.126	97.73
20.82	114958	0.150	0.137	97.48
20.90	110078	0.161	0.148	97.21
20.98	105405	0.175	0.160	96.92
21.07	100930	0.189	0.173	96.60
21.15	96645	0.203	0.186	96.26
21.23	92542	0.219	0.200	95.89
21.32	88614	0.235	0.215	95.50
21.40	84852	0.252	0.230	95.07
21.48	81250	0.271	0.248	94.62
21.57	77800	0.288	0.264	94.13
21.65	74498	0.309	0.283	93.62

File : A06.MWD

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Retention Time (min)	Molecular Weight	Intensity (mV)	$\frac{d(\text{Weight})}{d \log (\text{MW})}$	Weight %
21.73	71335	0.328	0.301	93.06
21.82	68307	0.350	0.320	92.47
21.90	65407	0.371	0.340	91.85
21.98	62630	0.394	0.361	91.18
22.07	59971	0.417	0.382	90.48
22.15	57426	0.441	0.404	89.74
22.23	54988	0.465	0.426	88.95
22.32	52653	0.489	0.448	88.13
22.40	50418	0.516	0.472	87.25
22.48	48278	0.542	0.495	86.34
22.57	46228	0.568	0.520	85.38
22.65	44266	0.596	0.546	84.37
22.73	42387	0.623	0.571	83.31
22.82	40587	0.650	0.595	82.21
22.90	38864	0.678	0.621	81.06
22.98	37214	0.706	0.647	79.86
23.07	35634	0.735	0.673	78.61
23.15	34122	0.763	0.699	77.31
23.23	32673	0.792	0.725	75.97
23.32	31286	0.820	0.751	74.57
23.40	29958	0.846	0.775	73.13
23.48	28686	0.874	0.800	71.64
23.57	27468	0.902	0.826	70.11
23.65	26302	0.929	0.850	68.52
23.73	25186	0.954	0.874	66.89
23.82	24116	0.979	0.896	65.22
23.90	23093	1.002	0.917	63.51
23.98	22112	1.022	0.936	61.76
24.07	21174	1.042	0.954	59.98
24.15	20275	1.059	0.970	58.16
24.23	19414	1.075	0.984	56.32
24.32	18590	1.087	0.995	54.45
24.40	17801	1.095	1.003	52.57
24.48	17045	1.101	1.008	50.67
24.57	16321	1.102	1.009	48.77
24.65	15629	1.102	1.009	46.87
24.73	14965	1.098	1.005	44.97
24.82	14330	1.089	0.998	43.09
24.90	13721	1.079	0.988	41.22
24.98	13139	1.065	0.975	39.37
25.07	12581	1.048	0.959	37.55
25.15	12047	1.028	0.941	35.76
25.23	11536	1.005	0.921	34.01
25.32	11046	0.980	0.897	32.31
25.40	10577	0.953	0.873	30.64
25.48	10128	0.926	0.848	29.03
25.57	9698	0.895	0.820	27.46
25.65	9286	0.865	0.792	25.95
25.73	8892	0.832	0.762	24.49

File : A06.MWD

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Retention Time (min)	Molecular Weight	Intensity (mV)	$\frac{d(\text{Weight})}{d \log (\text{MW})}$	Weight %
25.82	8515	0.800	0.733	23.09
25.90	8153	0.767	0.702	21.74
25.98	7807	0.734	0.672	20.45
26.07	7476	0.702	0.642	19.22
26.15	7158	0.670	0.613	18.04
26.23	6854	0.638	0.585	16.92
26.32	6563	0.607	0.556	15.85
26.40	6285	0.577	0.529	14.84
26.48	6018	0.548	0.501	13.87
26.57	5762	0.518	0.474	12.96
26.65	5518	0.490	0.449	12.09
26.73	5284	0.465	0.426	11.27
26.82	5059	0.439	0.402	10.50
26.90	4845	0.415	0.380	9.76
26.98	4639	0.391	0.358	9.07
27.07	4442	0.369	0.337	8.42
27.15	4253	0.345	0.316	7.81
27.23	4073	0.324	0.297	7.24
27.32	3900	0.304	0.278	6.70
27.40	3734	0.285	0.261	6.19
27.48	3576	0.267	0.244	5.72
27.57	3424	0.249	0.228	5.28
27.65	3279	0.232	0.213	4.87
27.73	3139	0.216	0.198	4.49
27.82	3006	0.201	0.184	4.13
27.90	2879	0.186	0.170	3.80
27.98	2756	0.172	0.158	3.49
28.07	2639	0.158	0.145	3.21
28.15	2527	0.147	0.134	2.95
28.23	2420	0.136	0.125	2.71
28.32	2317	0.126	0.115	2.48
28.40	2219	0.116	0.106	2.28
28.48	2125	0.108	0.098	2.08
28.57	2035	0.099	0.091	1.91
28.65	1948	0.091	0.084	1.75
28.73	1865	0.083	0.076	1.60
28.82	1786	0.076	0.070	1.46
28.90	1710	0.070	0.064	1.34
28.98	1638	0.065	0.059	1.22
29.07	1568	0.058	0.053	1.12
29.15	1502	0.053	0.048	1.02
29.23	1438	0.048	0.044	0.93
29.32	1377	0.045	0.041	0.85
29.40	1318	0.040	0.037	0.78
29.48	1262	0.036	0.033	0.72
29.57	1209	0.034	0.031	0.66
29.65	1158	0.030	0.028	0.60
29.73	1108	0.028	0.025	0.55
29.82	1061	0.024	0.022	0.51

Retention Time (min)	Molecular Weight	Intensity (mV)	$\frac{d(\text{Weight})}{d \log (\text{MW})}$	Weight %
29.90	1016	0.022	0.021	0.47
29.98	973	0.022	0.020	0.43
30.07	932	0.019	0.018	0.40
30.15	892	0.018	0.016	0.36
30.23	854	0.017	0.016	0.33
30.32	818	0.017	0.015	0.30
30.40	783	0.015	0.014	0.28
30.48	750	0.016	0.015	0.25
30.57	718	0.017	0.015	0.22
30.65	688	0.016	0.015	0.19
30.73	659	0.017	0.015	0.16
30.82	631	0.015	0.014	0.14
30.90	604	0.013	0.012	0.11
30.98	578	0.011	0.010	0.09
31.07	554	0.009	0.008	0.08
31.15	530	0.008	0.007	0.06
31.23	508	0.006	0.006	0.05
31.32	486	0.004	0.004	0.04
31.40	465	0.005	0.004	0.03
31.48	446	0.003	0.003	0.03
31.57	427	0.003	0.002	0.02
31.65	409	0.002	0.002	0.02
31.73	391	0.002	0.001	0.02
31.82	375	0.001	0.001	0.01
31.90	359	0.000	0.000	0.01
31.98	344	0.001	0.001	0.01
32.07	329	0.001	0.001	0.01
32.15	315	0.001	0.001	0.01
32.23	302	0.001	0.001	0.01
32.32	289	0.001	0.001	0.00
32.40	277	0.001	0.001	0.00
32.48	265	0.001	0.001	0.00
32.57	254	0.000	0.000	0.00
32.65	243	0.000	0.000	0.00
32.73	233	0.000	0.000	0.00
32.82	223	0.000	0.000	0.00
32.90	213	0.000	0.000	0.00
32.98	204	0.000	0.000	0.00

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Fig . 1

## Gel Permeation Chromatogram

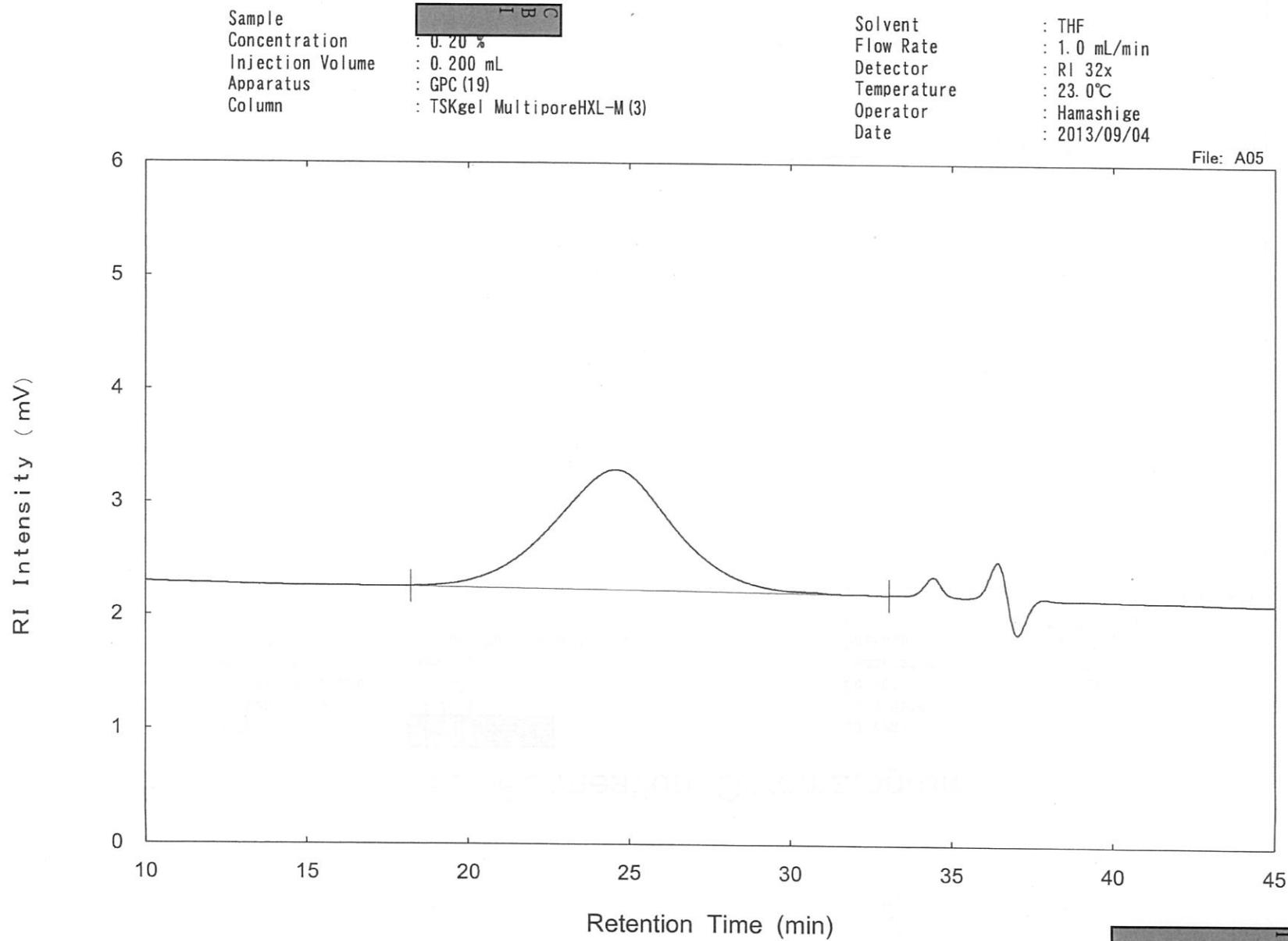


Fig . 2

## Gel Permeation Chromatogram

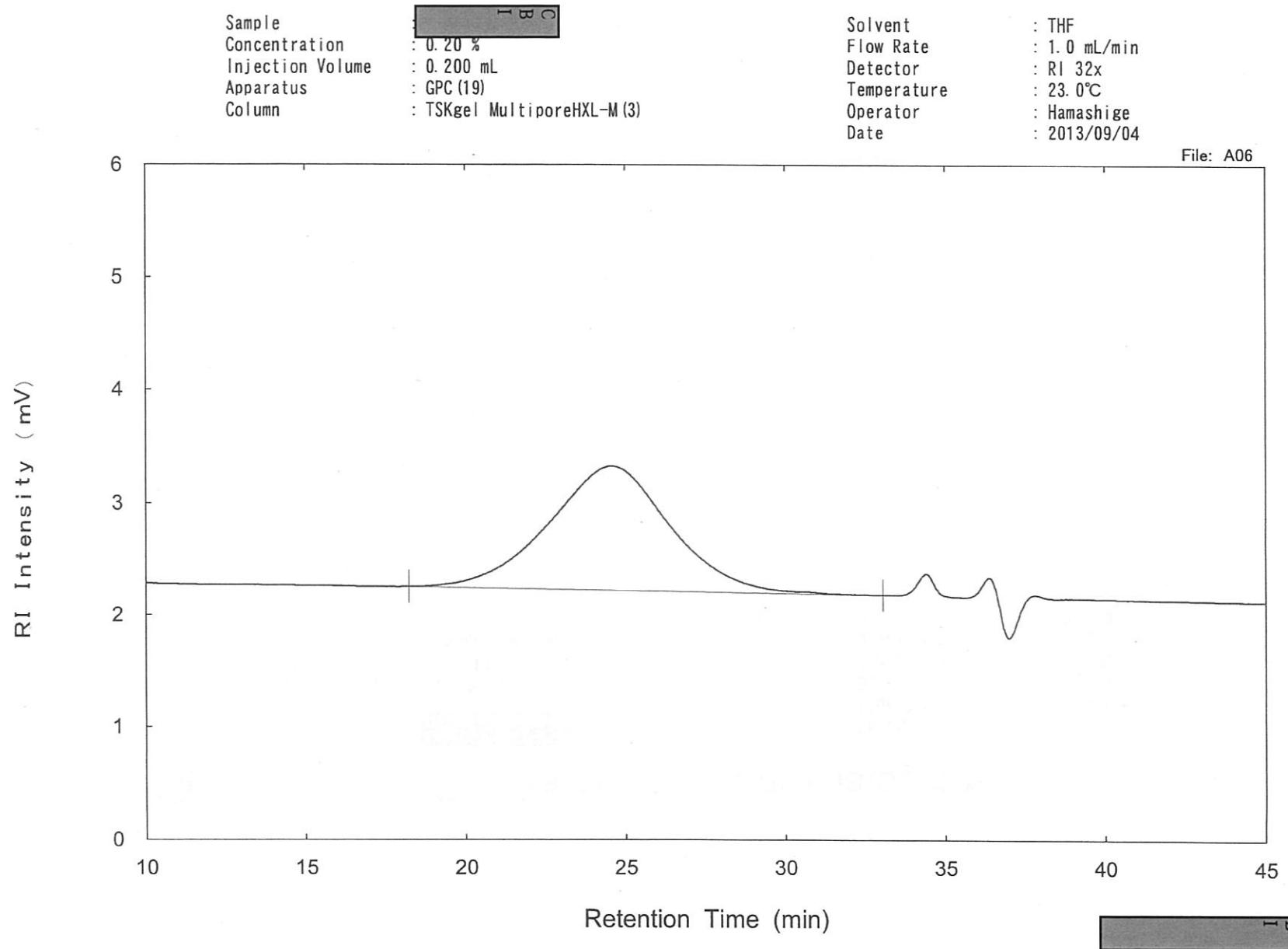


Fig . 3

## Gel Permeation Chromatogram

Sample : Solvent  
Concentration : -  
Injection Volume : 0.200 mL  
Apparatus : GPC (19)  
Column : TSKgel MultiporeHXL-M (3)

Solvent : THF  
Flow Rate : 1.0 mL/min  
Detector : RI 32x  
Temperature : 23.0°C  
Operator : Hamashige  
Date : 2013/09/04

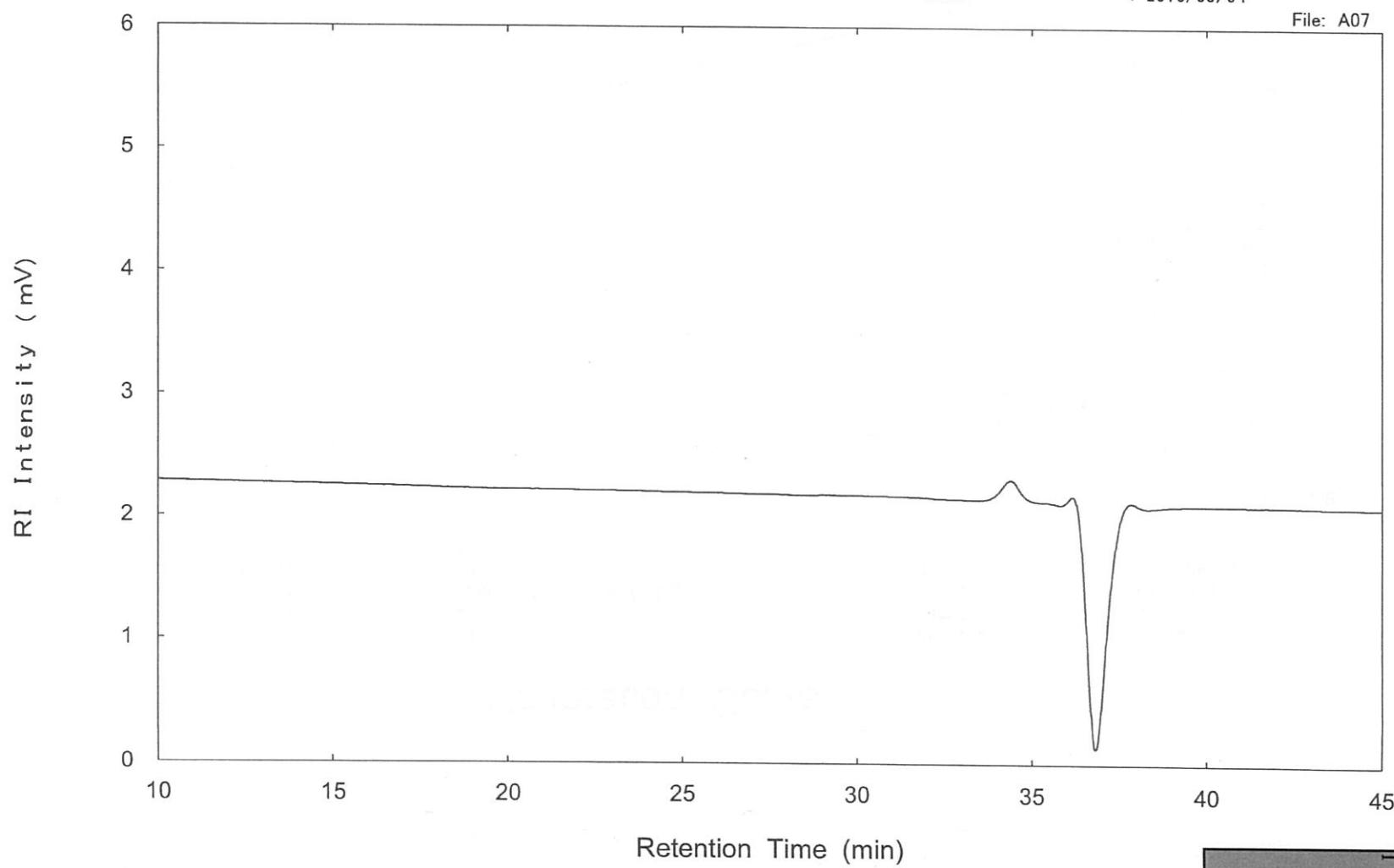


Fig . 4

## Calibration Curve

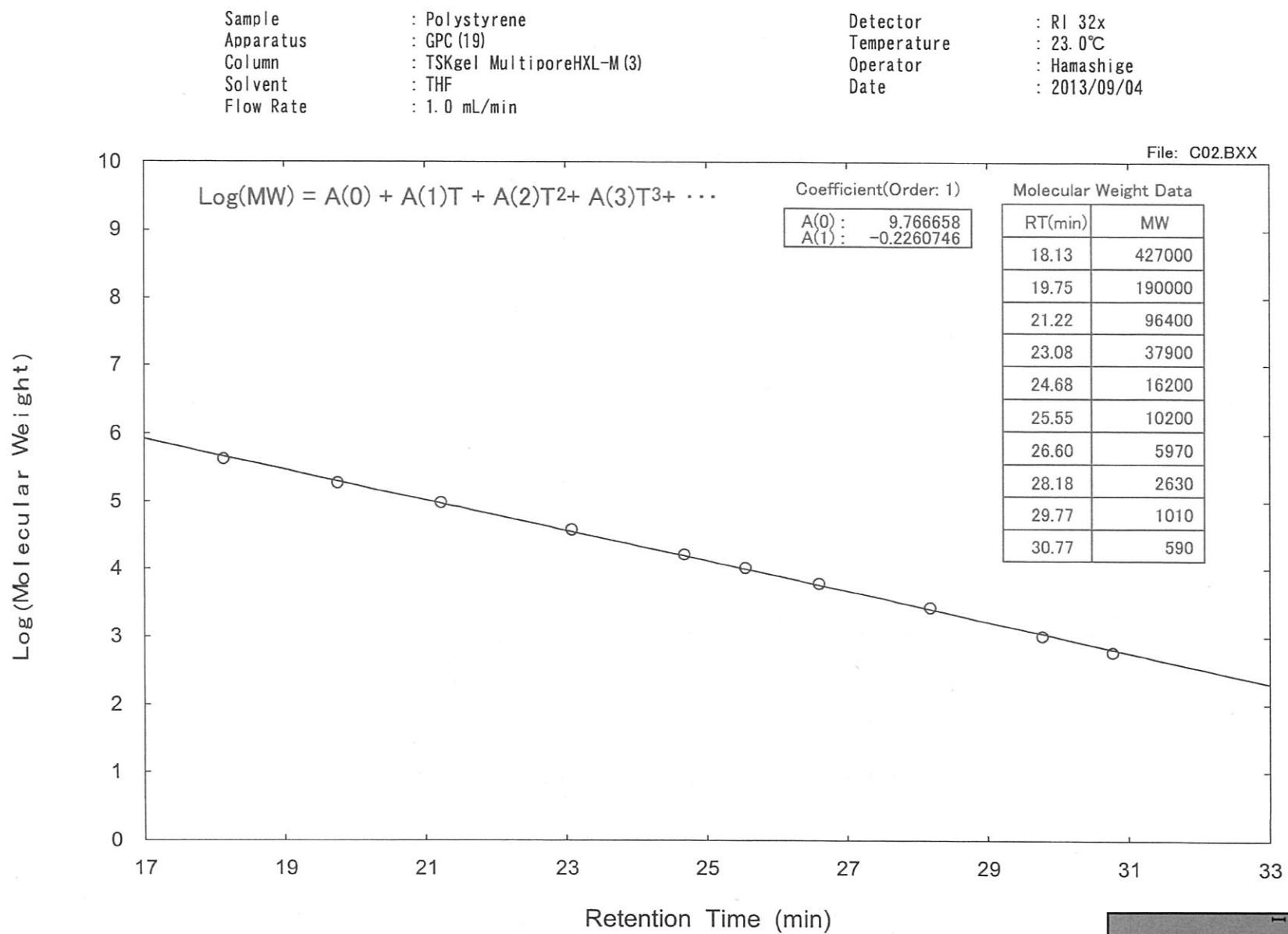


Fig. 5

## Molecular Weight Distribution Curve

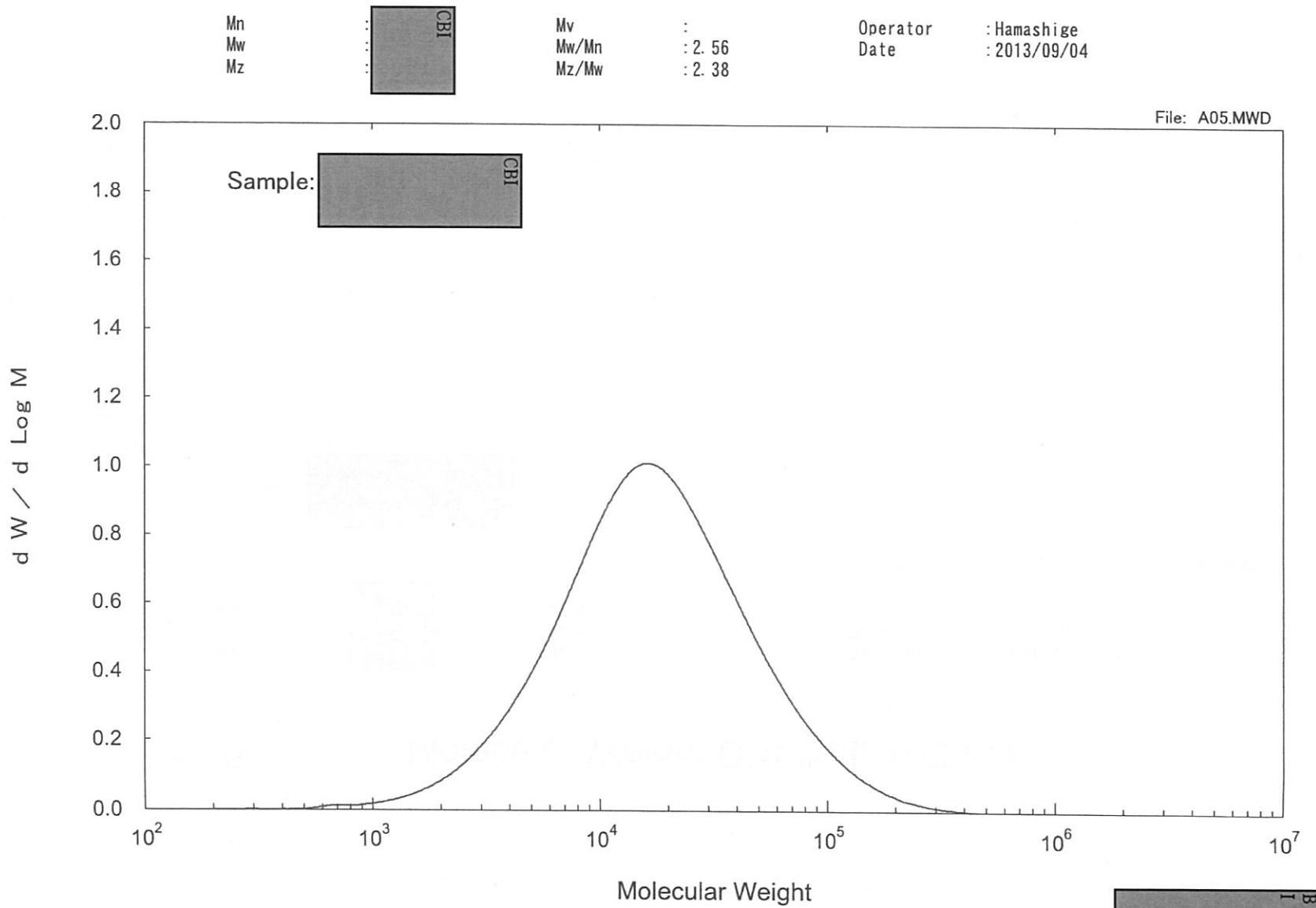


Fig . 6

## Molecular Weight Distribution Curve

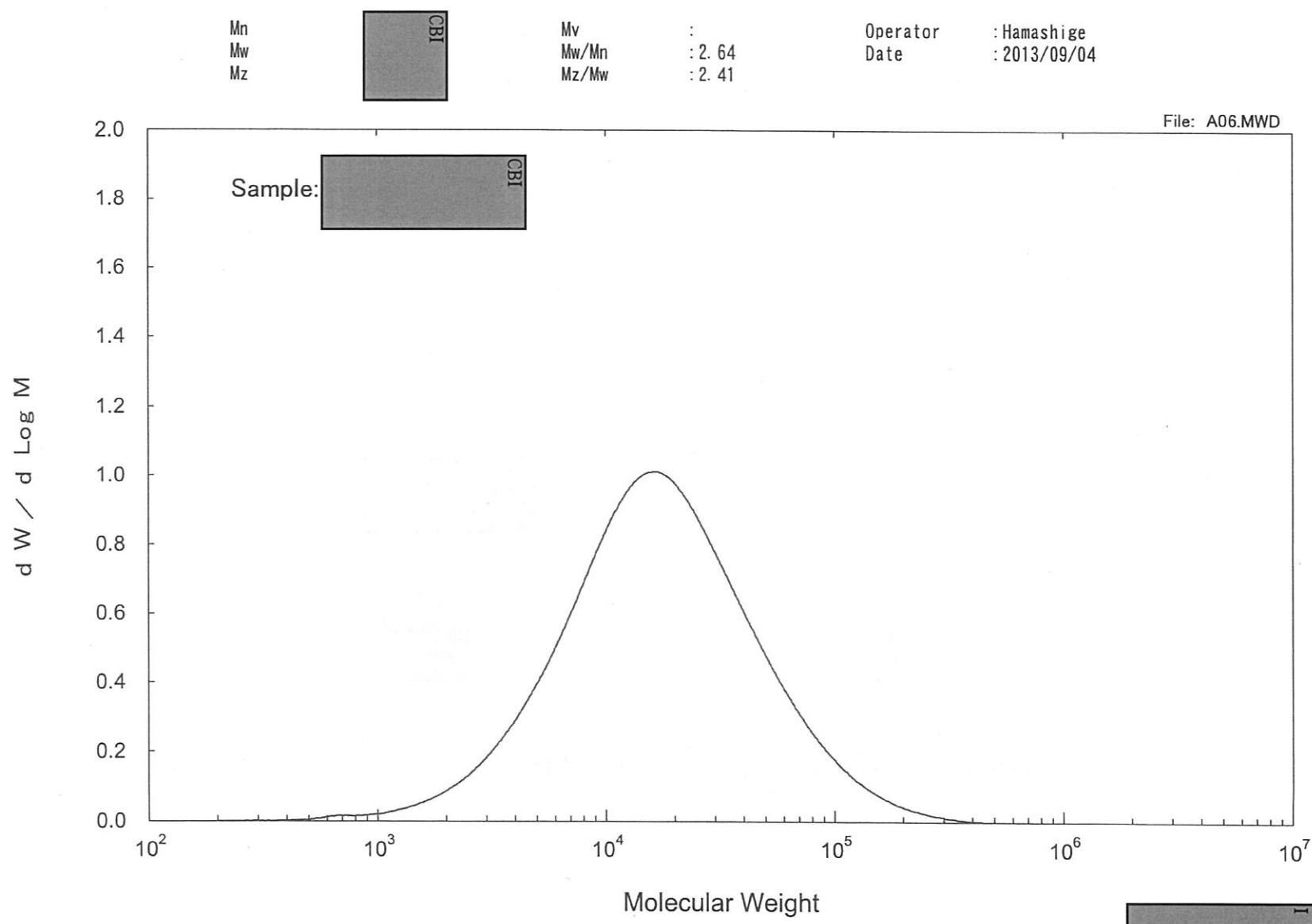


Fig . 7

## Molecular Weight Distribution Curve

